

Graphene Based Ultra-Capacitors for Safer, More Efficient Energy Storage

Luke B. Roberson, Paul J. Mackey, Carson J. Zide

*National Aeronautics and Space Administration, Kennedy Space Center, FL 32899
Chemical Engineering Major at University of South Florida*

Abstract

Current power storage methods must be continuously improved in order to keep up with the increasingly competitive electronics industry. This technological advancement is also essential for the continuation of deep space exploration. Today's energy storage industry relies heavily on the use of dangerous and corrosive chemicals such as lithium and phosphoric acid. These chemicals can prove hazardous to the user if the device is ruptured. Similarly they can damage the environment if they are disposed of improperly. A safer, more efficient alternative is needed across a wide range of NASA missions. One solution would be a solid-state carbon based energy storage device. Carbon is a safer, less environmentally hazardous alternative to current energy storage materials. Using the amorphous carbon nanostructure, graphene, this idea of a safer portable energy is possible. Graphene was electrochemically produced in the lab and several coin cell devices were built this summer to create a working prototype of a solid-state graphene battery.

Background

The process of electrochemical exfoliation of graphite uses graphite foil as an anode and a platinum wire as the cathode. The distance between the two electrodes is kept constant at 2 cm throughout the exfoliation process. Applying voltage results in the reduction of water at the cathode, producing hydroxyl ions that are strongly nucleophilic in the electrolyte. These nucleophilic Hydroxyl ions attack and oxidize the graphite starting at the edges and grain boundaries. This leads to depolarization and expansion of the layers, which accelerates the intercalation of sulfate ions and water molecules between the layers of graphite. The resulting reduction and oxidation of these intercalated molecules during the electrochemical process causes gasses to form between layers. These gasses can apply forces to the graphite layers strong enough to separate them from each other. The product of the exfoliation is filtered, collected and rinsed with deionized water using vacuum filtration.

Materials

- Deionized water
- $(\text{NH}_4)_2\text{SO}_4$ (Ammonium Sulfate)
- 4.5 cm x 2 cm sheet of Graphite foil
- Platinum wire
- PVC separator membrane

- Coin cell assembly parts
- Sulfuric acid

Equipment

- 2 – 400 mL beakers
- 250 mL beaker
- Centigram scale
- Constant 10 volt power source
- PTFE membrane filter with 0.2 μm pore size
- Filter flask
- Buchner funnel
- Tubing
- Filter paper
- Hydraulic crimping press
- Disposable dropper
- Vacuum pump
- Tweezers

Method

1. Measure out 100 ml of $(\text{NH}_4)_2\text{SO}_4$ and add it to a 250 ml beaker.
2. Fix the negative electrical lead to the platinum wire to form the cathode.
3. Cut the graphite foil diagonally and over lay the two halves symmetrically.
4. Fix the positive electrical lead to the longest point (seen in figure 1) of graphite foil to form the anode.

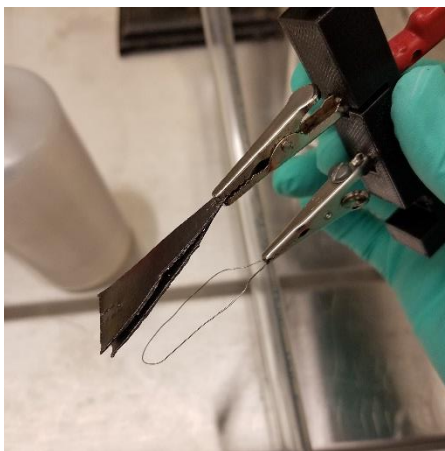


Figure 1: Graphite foil is cut, overlaid and attached to the electrode holder.

5. Attach both electrodes exactly 2 cm apart on the electrode holder (seen in figure 2).

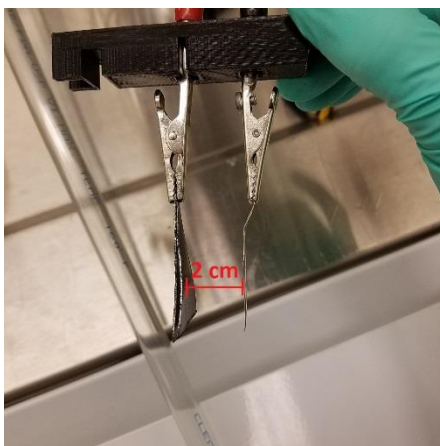


Figure 2: Electrodes are evenly spaced by 2 cm.

6. Lower the two electrodes into the beaker until the graphite foil is almost completely submerged in the solution. (Figure 3)

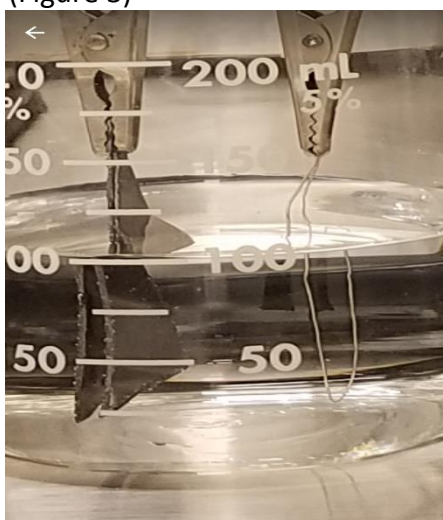


Figure 3: Electrodes submerged in electrolyte

7. Apply a positive voltage of 10 volts to the graphite electrode to initiate the electrochemical exfoliation process for 10 minutes.
8. Remove electrodes from solution.
9. Prepare to filter your sample by placing a PTFE membrane filter with 0.2 μm pore size in the Buchner funnel.
10. Wet the filter paper in the Buchner funnel with clean solvent (deionized water).
11. Slowly pour sample into the center of the filter paper.
12. Rinse beaker with clean solvent to collect residual solid.
13. Pour solvent/ residual solid through filter.
14. Continue to rinse solid with 50 mL of clean solvent to remove residual salts.
15. Remove paste from the filter paper and add it to a 250 ml beaker.
16. Place beaker with paste in the dry box and let dry for 3 days to eliminate all residual DI water.
17. Break up graphene flakes in a sealed bag until nearly powder.

Coin Cell Assembly

1. Place coin cell cup on glass dish.
2. Punch 19mm PVC separator membrane.
3. Measure out .01 grams of graphene flakes.
4. Place .005 grams of graphene paste in center of cell cup.
5. Add 1 drop of sulfuric acid on the graphene.
6. Punch 19mm PVC separator
7. Soak 19mm PVC separator in sulfuric acid
8. Place 19mm PVC separator over the graphene.
9. Place .005 grams of graphene paste on separator.
10. Add 1 drop of sulfuric acid on graphene.
11. Place cell cap directly on top of the graphene.
12. Crimp the coin cell on hydraulic crimping press.
13. Place finished cell in charge/discharge tester.
14. Record current vs. potential data.

Results

After several different coin cell assembly methods were tested, the assembly method above was chosen as the most efficient and reproducible. Five more coin cells were assembled using this method and tested to have consistent current vs. potential data. An average data set for these five graphene energy storage cells being charged from 0 Volts to .7 Volts then discharged back down to 0 volts is shown in Figure 4.

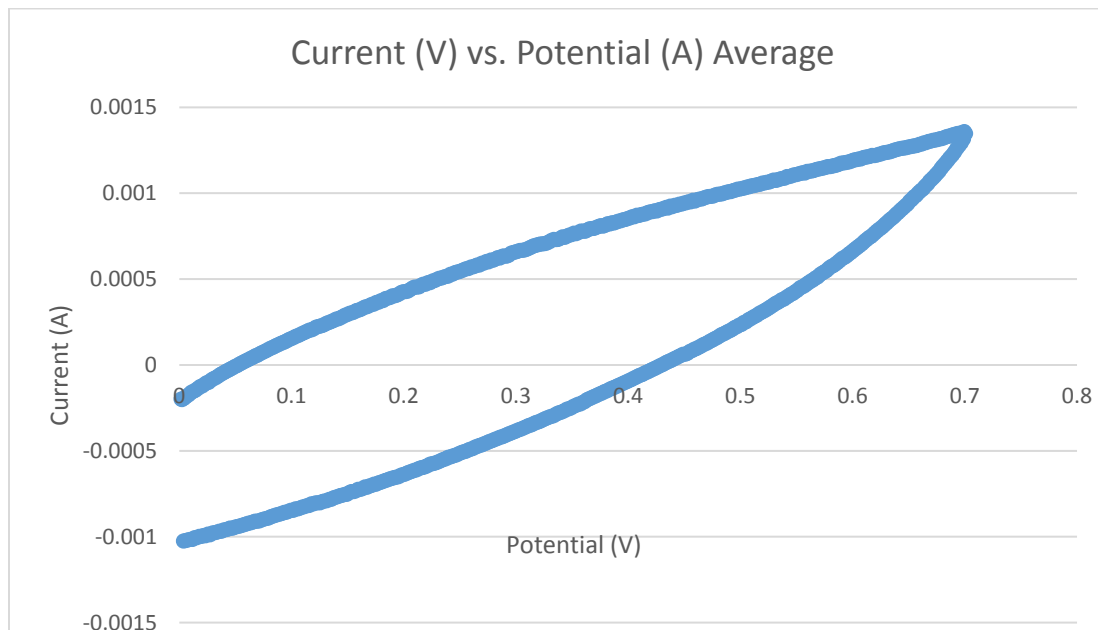


Figure 4: Average current vs. potential of graphene based energy storage device.

Future Study

Future research in this study would explore the forging of aluminum and copper electrodes coated with a layer of graphene glue made up of a poly-vinyl, a solvent and graphene paste. This may lead to a way of keeping the layers of graphene separate in an ionic solid solution which would allow more surface area contact to all of the graphene sheets. This would mean that a significant amount more of exposed charge storage area would be available and used.

References:

Parvez, Khaled, Zhong-Shuai Wu, Rongjin Li, Xianjie Liu, Robert Graf, Xinliang Feng, and Klaus Müllen. "Exfoliation of Graphite into Graphene in Aqueous Solutions of Inorganic Salts." *J. Am. Chem. Soc. Journal of the American Chemical Society* 136.16 (2014): 6083-091. Web.

....